Federal Democratic Republic of Ethiopia

☞ EDICT OF GOVERNMENT ☜

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ET ISO 3707 (1976) (English): Phosphoric acid for industrial use (including foodstuffs) -- Determination of calcium content -- Flame atomic absorption method

ISO INSIDE

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ETHIOPIAN STANDARD

ES :

First edition

Edible salt— Specification

ICS: 67.220.20

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Foreword

This Ethiopian Standard has been prepared under the direction of the Technical Committee for Food additives and contaminants (TC 21) and published by the Ethiopian Standards Agency (ESA).

The standard is identical with ISO 3707:1976 “Determination of Calcium content - Flame atomic absorption method ”, published by the International Organization for Standardization (ISO).

For the purpose of this Ethiopian Standard, the adopted text shall be modified as follows.

- The phrase “International Standard” shall be read as “Ethiopian Standard”; and
- A full stop (.) shall substitute comma (,) as decimal marker.

Phosphoric acid for industrial use (including foodstuffs) – Determination of calcium content – Flame atomic absorption method

1 SCOPE

This International Standard specifies a flame atomic absorption method for the determination of the calcium content of phosphoric acid for industrial use (including foodstuffs).

2 FIELD OF APPLICATION

The method is applicable to products having a calcium content higher than 50 mg/kg but, by preparation of a suitable calibration graph, the range can be extended down to 10 mg/kg.

Soluble SiO_2 present in the test solution at levels lower than 5 µg/ml does not interfere.

2.1 Special case

Presence of soluble SiO_2 at levels higher than 5 µg/ml of test solution (under study).

3 PRINCIPLE

Addition, to a hydrochloric acid solution of the test portion, of sodium ions to promote and to stabilize the emission of calcium, and of lanthanum ions to suppress the interference of aluminium. Aspiration of the solution into an acetylene-dinitrogen monoxide flame and determination of the calcium content by photometric measurement of the absorption of the 422,7 nm line emitted by a hollow-cathode calcium lamp.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only water doubly distilled in borosilicate glass apparatus with ground glass joints, or water of equivalent purity.

4.1 Phosphoric acid, 40 g/l solution free from calcium.

Weigh, to the nearest 0,1 g, 29 g of phosphorus(V) oxide (P_2O_5) and spread out in a shallow layer in a suitable dish. Allow the dish to stand in a closed vessel containing water (for example a desiccator containing water in place of a desiccant), in order to effect the initial hydration. Then dissolve the hydrated oxide in 1 000 ml of water.

4.2 Hydrochloric acid, approximately 6 N solution.

4.3 Sodium chloride and lanthanum chloride, combined solution.

Dissolve 25,5 g of sodium chloride and 10 g of lanthanum chloride heptahydrate ($LaCl_3.7H_2O$) in water and dilute to 100 ml.

1 ml of this solution contains approximately 100 mg of Na and 100 mg of lanthanum chloride heptahydrate.

4.4 Calcium, standard solution, corresponding to 1,000 g of Ca per litre.

Weigh, to the nearest 0,000 1 g, 2,497 2 g of calcium carbonate, previously dried at 250 °C for 2 h and cooled in a desiccator. Place in a beaker of convenient capacity (for example 600 ml) and dissolve carefully in 30 ml of the hydrochloric acid solution (4.2). Dilute the solution and transfer quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 1,000 mg of Ca.

Store this solution in a bottle of material free from calcium.

4.5 Calcium, standard solution, corresponding to 0,050 g of Ca per litre.

Transfer 50,0 ml of the standard calcium solution (4.4) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 50 µg of Ca.

Prepare this solution just before use.

5 APPARATUS

Ordinary laboratory apparatus, of material free from calcium, and

5.1 Atomic absorption spectrophotometer, fitted with a burner fed with acetylene and dinitrogen monoxide.

5.2 Hollow-cathode calcium lamp.

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6 PROCEDURE

6.1 Test portion

Weigh, by difference, to the nearest 0,001 g, about 5 g of the test sample.

6.2 Preparation of the calibration graph

6.2.1 *Preparation of the standard matching solutions*

Into each of a series of five 100 ml one-mark volumetric flasks, place a quantity of the phosphoric acid solution (4.1) such that it contains the same quantity of P_2O_5 as the test portion (6.1). Add 1 ml of the combined solution (4.3) and 4 ml of the hydrochloric acid solution (4.2) and then, respectively, the volumes of the standard calcium solution (4.5) shown in the following table.

Standard calcium solution (4.5)	**Corresponding mass of Ca**
ml	µg
0*	0
1,0	50
2,0	100
4,0	200
6,0	300

* Blank test on reagents of the calibration graph.

Dilute the contents of each flask to the mark and mix.

NOTE — If the test solution contains less than 50 µg of calcium (Ca) in 100 ml, prepare a more dilute standard calcium solution by diluting 10,0 ml of the standard calcium solution (4.5) to 100 ml. Use this weaker solution to prepare a calibration graph covering the range 0 to 50 µg of Ca in 100 ml. The bracketing measurements (6.3.2.2) should then be carried out between two standard matching solutions differing by 5 µg of Ca in 100 ml.

6.2.2 *Spectrophotometric measurements*

6.2.2.1 ADJUSTMENT OF THE APPARATUS FITTED WITH THE HOLLOW CATHODE CALCIUM LAMP (5.2)

Switch on the current to the apparatus (5.1) a sufficient time in advance to ensure stabilization. Adjust the wavelength to about 422,7 nm and adjust the sensitivity and the aperture of the slit according to the characteristics of the apparatus. Adjust the pressure of the acetylene and of the dinitrogen monoxide according to the characteristics of the aspirator burner. Adjust the aspiration rate to between 2 and 4 ml/min.

6.2.2.2 MEASUREMENTS

Aspirate the series of standard matching solutions (6.2.1) in the flame and measure the absorbance for each. Take care to keep the aspiration rate constant throughout the preparation of the calibration graph.

Aspirate water through the burner after each measurement.

6.2.3 *Plotting the calibration graph*

Plot a graph having, for example, the numbers of micrograms of Ca contained in 100 ml of the standard matching solutions as abscissae and the corresponding values of the absorbances, reduced by the value for the standard matching solution No. 0, as ordinates.

6.3 Determination

6.3.1 *Preparation of the test solution*

Transfer the test portion (6.1) to a 500 ml one-mark volumetric flask and dilute to about 250 ml. Add 20 ml of the hydrochloric acid solution (4.2), and 5 ml of the combined solution (4.3). Dilute to the mark and mix.

If the calcium content is between 50 and 200 mg/kg, carry out the measurements directly on the test solution thus obtained.

If the calcium content is higher, carry out further dilutions as indicated in the following table.

Expected Ca content	**Aliquot portion of the test solution (6.3.1) to be taken**	**Combined solution (4.3) to be added**	**Final volume of the solution**
mg/kg	ml	ml	ml
200 to 500	50	0,50	100
500 to 1 000	25	0,75	100
1 000 to 1 500	20	0,80	100
1 500 to 2 000	10	0,90	100

6.3.2 *Spectrophotometric measurements*

6.3.2.1 PRELIMINARY MEASUREMENT

Carry out a preliminary measurement on the test solution (6.3.1) following the procedure specified in 6.2.2.2, at the same time as the spectrophotometric measurements are carried out on the standard matching solutions (6.2.1).

From the calibration graph (6.2.3), calculate the approximate concentration of Ca, in micrograms per 100 ml of the test solution (6.3.1).

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6.3.2.2 BRACKETING MEASUREMENT

Carry out a second measurement on the test solution (6.3.1) by bracketing between two standard matching solutions differing by only 25 μg of Ca in 100 ml.

To prepare these standard matching solutions, follow the procedure specified in 6.2.1, using, however, suitable quantities of the standard calcium solution (4.5).

7 EXPRESSION OF RESULTS

The concentration C of calcium, expressed as micrograms of Ca per 100 ml of the test solution, is given by the formula

$$C = C_1 + (C_2 - C_1)\frac{A_0 - A_1}{A_2 - A_1}$$

where

C_1 is the concentration, in micrograms per 100 ml, of the weaker bracketing solution;

A_1 is the corresponding value of the absorbance;

C_2 is the concentration, in micrograms per 100 ml, of the stronger bracketing solution;

A_2 is the corresponding value of the absorbance;

A_0 is the value of the absorbance corresponding to the test solution (6.3.1).

The calcium (Ca) content, expressed in milligrams per kilogram, is given by the formula

$$\frac{C}{m} \times \frac{500}{100} \times D = \frac{C}{m} \times 5 \times D$$

where

C is the concentration of Ca, expressed as micrograms per 100 ml of the test solution;

m is the mass, in grams, of the test portion (6.1);

D is the dilution ratio (see the table in 6.3.1);

5 is the ratio of the volume of the test solution (6.3.1) to the volume of the calibration solutions (6.2.1).

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.

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ANNEX

ISO PUBLICATIONS RELATING TO PHOSPHORIC ACID FOR INDUSTRIAL USE

ISO 847 – Determination of sulphate content – Titrimetric method.

ISO 848 – Determination of calcium content – Titrimetric method.

ISO 849 – Determination of iron content – 2,2′-Bipyridyl spectrophotometric method.

ISO 2997 – Determination of sulphate content – Method by reduction and titrimetry.

ISO 3359 – Determination of arsenic content – Silver diethyldithiocarbamate photometric method.

ISO 3360 – Determination of fluorine content – Alizarin complexone and lanthanum nitrate photometric method.*

ISO 3361 – Determination of soluble silica content – Reduced molybdosilicate spectrophotometric method.

ISO 3706 – Determination of total phosphorus(V) oxide content – Quinoline phosphomolybdate gravimetric method.*

ISO 3707 – Determination of calcium content – Flame atomic absorption method.*

ISO 3708 – Determination of chloride content – Potentiometric method.*

ISO 3709 – Determination of nitrogen oxides content – 3,4-Xylenol spectrophotometric method.*

ISO 4285 – Sampling technique.

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* Also applicable to phosphoric acid for use in the foodstuffs industry.

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Ethiopian Standards Agency

Organization and Objectives

The Ethiopian Standards Agency (ESA) is the national standards body of Ethiopia established in 2010 based on regulation No. 193/2010.ESA is established due to the restructuring of Quality and Standards Authority of Ethiopia (QSAE) which was established in 1970.

ESA's objectives are:-

- Develop Ethiopian standards and establish a system that enable to check weather goods and services are in compliance with the required standards,
- Facilitate the country's technology transfer through the use of standards,
- Develop national standards for local products and services so as to make them competitive in the international market.

Ethiopian Standards

The Ethiopian Standards are developed by national technical committees which are composed of different stakeholders consisting of educational Institutions, research institutes, government organizations, certification, inspection, and testing organizations, regulatory bodies, consumer association etc. The requirements and/ or recommendations contained in Ethiopian Standards are consensus based that reflects the interest of the TC representatives and also of comments received from the public and other sources. Ethiopian Standards are approved by the National Standardization Council and are kept under continuous review after publication and updated regularly to take account of latest scientific and technological changes.
Orders for all Ethiopian Standards, International Standard and ASTM standards, including electronic versions, should be addressed to the Documentation and Publication Team at the Head office and Branch (Liaisons) offices. A catalogue of Ethiopian Standards is also available freely and can be accessed in from our website.

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International Involvement
ESA, representing Ethiopia, is a member of the International Organization for Standardization (ISO), and Codex Alimentarius Commission (CODEX). It also maintains close working relations with the international Electro-technical Commission (IEC) and American Society for Testing and Materials (ASTM).It is a founding member of the African Regional Organization for standardization (ARSO).

More Information?
Contact us at the following address.
The Head Office of ESA is at Addis Ababa.

☎ 011- 646 06 85, 011- 646 05 65
📠 011-646 08 80
✉ 2310 Addis Ababa, Ethiopia
E-mail: info@ethiostandards.org,
Website: www.ethiostandards.org

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